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(54) **LUBRICATING OIL COMPOSITION FOR STEPLESS TRANSMISSIONS AND METHOD FOR LUBRICATING STEPLESS TRANSMISSIONS THEREWITH**

(57) A lubricant composition for continuous variable transmissions which comprises a base oil and auxiliaries incorporated therein which are a sulfur-based extreme pressure additive (A), a phosphorus-based extreme pressure additive (B), and an alkaline earth metal-based detergent (C).

It is superior in wear resistance and extreme pressure properties and keeps the coefficient of friction high for a long period of time, so that it is capable of transmitting a large capacity of torque when it is applied to an continuous variable transmission. It is particularly suitable for a transmission of metal belt type.

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Description**Technical Field**

5 The present invention relates to a lubricant composition for continuous variable transmissions and also to a method for lubricating continuous variable transmissions with said lubricant composition. This lubricant composition is superior in wear resistance and extreme pressure properties and is capable of keeping the coefficient of friction high for a long period of time and of transmitting a large amount of torque. It is particularly suitable for transmissions of metal belt type.

10 Background Art

The conventional automotive automatic transmission consists of a torque converter and a finitely variable transmission comprising several gear trains. The transmission of this type has a problem with low efficiency due to slip loss in the torque converter and torque loss at the time of speed change. To address this problem, there has recently been 15 developed an automotive continuous variable transmission that employs a steel belt. It is now in practical use.

This transmission, however, suffers the disadvantage that the coefficient of friction decreases so much under a high load that it cannot transmit a large torque and the belt slips at the time of rapid acceleration, resulting in a low ratio of torque transmission. This disadvantage arises from the fact that it is lubricated with the conventional lubricant (so-called ATF) for finitely variable transmissions. For this reason, the above-mentioned continuous variable transmission is used 20 only for automobiles with a small-capacity engine (generating a small torque).

With a view to overcoming this disadvantage, attempts have been made to improve the ratio of torque transmission from the standpoint of mechanism. However, it has been found that improvement in torque transmission is incompatible with improvement in wear resistance, because torque transmission is always accompanied by slight slipping (which is inherent in the mechanism employed).

25 It is an object of the present invention to provide a lubricant composition and a method for lubrication with said lubricant composition. The lubricant composition of the present invention is superior in wear resistance and extreme pressure properties, capable of keeping a coefficient of friction high for a long period of time, and capable of transmitting a large amount of torque. It is particularly suitable for transmissions of metal belt type.

30 Disclosure of the Invention

The present inventors carried out a series of researches to develop a lubricant composition for continuous variable transmissions which meets the above-mentioned requirements. As a result, it was found that a lubricant keeps the coefficient of friction higher than 0.10 for a long period time if its base oil is incorporated with a sulfur-based extreme pressure additive, a phosphorus-based extreme pressure additive, and an alkaline earth metal-based detergent as 35 essential ingredients. This finding led to the present invention.

It is an object of the present invention to provide a lubricant composition for continuous variable transmissions which comprises a base oil, a sulfur-based extreme pressure additive (A), a phosphorus-based extreme pressure additive (B), and an alkaline earth metal-based detergent (C).

40 It is another object of the present invention to provide a method for lubricating continuous variable transmissions with said lubricant composition.

The preferred embodiments of the present invention are as follows.

- 45 • A lubricant composition for continuous variable transmissions as defined above, wherein the sulfur-based extreme pressure additive is at least one species selected from sulfurized oils and fats, thiocarbamates, and thioterpenes.
- A lubricant composition for continuous variable transmissions as defined above, wherein the phosphorus-based extreme pressure additive is at least one species selected from tricresyl phosphate and amine salts of alkyl or alkenyl acid phosphate ester.
- 50 • A lubricant composition for continuous variable transmissions as defined above, wherein the alkaline earth metal-based detergent is calcium phenate.
- A lubricant composition for continuous variable transmissions as defined above, wherein the amount of components (A), (B), and (C) based on the total amount of the lubricant composition is 0.05-5 wt%, 0.05-5 wt%, and 0.05-8 wt%, respectively.
- 55 • A lubricant composition for continuous variable transmissions as defined above, wherein the continuous variable transmission is of metal belt type.
- A method for lubricating continuous variable transmissions with the lubricant composition defined above.
- A method for lubricating continuous variable transmissions, designed for performing continuous speed change and torque transmission simultaneously, with the lubricant composition defined above.

Best Mode of Carrying out the Invention

5 The lubricant composition of the present invention is prepared usually from a mineral oil or synthetic oil as the base oil which is not specifically restricted in kind and properties. Preferred base oils are those which have a kinematic viscosity (at 100°C) of 1-50 cSt, preferably 2-15 cSt, a value of %C_A (ASTM D3238-80) smaller than 20, preferably smaller than 10, and a pour point of lower than -10 °C, preferably lower than -15°C.

10 Examples of the mineral oil include paraffin oil, intermediate oil, and naphthene oil, which are obtained by the ordinary refining process such as solvent extraction and hydrogenation. Of these examples, paraffin oil is particularly preferable.

15 Examples of the synthetic oil include polybutene, polyolefins (such as α -olefin homopolymer and copolymer like ethylene- α -olefin copolymer), esters (such as polyol ester, dibasic acid ester, and phosphoric ester), ethers (such as polyphenyl ether), polyglycol, alkylbenzene, and alkynaphthalene. Of these examples, polyolefins and polyol esters are preferable.

20 The above-mentioned mineral oils and synthetic oils may be used alone or in combination with one another as the base oil.

25 The lubricant composition of the present invention contains a sulfur-based extreme pressure additive as the component (A), which is not specifically restricted so long as it has sulfur in the molecule and is capable of dissolving or uniformly dispersing in the base oil to exhibit the extreme pressure properties and good wear resistance. It includes, for example, sulfurized vegetable and animal oils and synthetic oils, olefin polysulfide, dihydrocarbyl polysulfide, sulfurized mineral oils, thiocarbamates, thioterpenes, and dialkyl thiodipropionates.

30 Examples of the sulfurized vegetable and animal oils include sulfurized lard, sulfurized rapeseed oil, sulfurized castor oil, sulfurized soybean oil, sulfurized rice bran oil, disulfurized fatty acids (such as sulfurized oleic acid), and sulfurized esters (such as sulfurized methyl oleate). Olefin polysulfides are obtained by reacting C₃₋₂₀ olefins or its oligomer with a sulfurizing agent. The preferable examples of the olefin include propylene, isobutene, and diisobutene.

35 The examples of the sulfurizing agent include sulfur and sulfur halide such as sulfur chloride.

40 The dihydrocarbyl polysulfide is a compound represented by the formula (I) below.



45 (where R¹ and R² each denotes a C₁₋₂₀ alkyl group, a C₆₋₂₀ aryl group, a C₇₋₂₀ alkylaryl group, or a C₇₋₂₀ arylalkyl group (which may be the same or different), and x is a real number (or a rational number) of 2-8.)

50 Examples of the groups represented by R¹ and R² in the formula (I) above include methyl group, ethyl group, n-propyl group, isopropyl group, n-butyl group, isobutyl group, sec-butyl group, t-butyl group, pentyl groups, hexyl groups, heptyl groups, octyl groups, nonyl groups, decyl groups, dodecyl groups, cyclohexyl group, cyclooctyl group, phenyl group, naphthyl group, tolyl group, xylyl group, benzyl group, and phenethyl group.

55 Preferred examples of the dihydrocarbyl polysulfide include dibenzyl polysulfide, di-t-nonylpolysulfide, and didodecyl polysulfide.

60 Examples of the thiocarbamates include zinc dithiocarbamate. Examples of the thiopertene include a reaction product of pinene and phosphorus pentasulfide. Examples of the dialkyl thiodipropionate include dilauryl thiodipropionate and distearyl thiodipropionate. Of these inert extreme pressure additive such as sulfurized oils, thiocarbamates, and thioterpenes are preferable in terms of extreme pressure properties and wear resistance.

65 In the present invention, the above-mentioned sulfur-based extreme pressure additives may be used alone or in combination with one another. Their amount should be 0.05-5 wt% of the total amount of the lubricant composition. An amount less than 0.05 wt% is not enough for sufficient extreme pressure performance and wear resistance. An amount exceeding 5 wt% produces an adverse effect on the oxidative stability. A preferred amount (from the standpoint of extreme properties, wear resistance, and oxidative stability) is 0.1-3 wt% of the total amount of the lubricant composition.

70 The lubricant composition of the present invention contains a phosphorus-based extreme pressure additive as the component (B), which is not specifically restricted so long as it has phosphorus in the molecule and is capable of dissolving or uniformly dispersing in the base oil to exhibit the extreme pressure properties and good wear resistance. It includes, for example, phosphate ester, acid phosphate ester, phosphite ester, acid phosphite ester, thiophosphate ester, acid thiophosphate ester, amine salts thereof, and phospho-sulfurized terpenes (such as reaction products of pinene and phosphorus pentasulfide).

75 Examples of the phosphate ester and phosphite ester include tributyl phosphate and phosphite, trihexyl phosphate and phosphite, tri-2-ethylhexyl phosphate and phosphite, tridecyl phosphate and phosphite, triauryl phosphate and phosphite, trimyristyl phosphate and phosphite, tripalmityl phosphate and phosphite, tristearyl phosphate and phosphite, trioleyl phosphate and phosphite, and other C₃₋₃₀ alkyl or alkenyl phosphate or phosphite esters; and triphenyl phosphate and phosphite, tricresyl phosphate and phosphite, and other C₆₋₃₀ aryl phosphate or phosphite esters.

80 Examples of the acid phosphate or phosphite ester include mono- or dibutyl hydrogen phosphate and phosphite,

5 mono- or dipentyl hydrogen phosphate and phosphite, mono- or di-2-ethylhexyl hydrogen phosphate and phosphite, mono- or dipalmityl hydrogen phosphate and phosphite, mono- or dilauryl hydrogen phosphate and phosphite, mono- or distearyl hydrogen phosphate and phosphite, mono- or dioleyl hydrogen phosphate and phosphite, and other C₃-30 alkyl or alkenyl acid phosphate and phosphite; and mono- or diphenyl hydrogen phosphate and phosphite, mono- or dicresyl hydrogen phosphate and phosphite, and other C₆-30 aryl acid phosphate and phosphite.

10 Examples of the thiophosphate ester and thiophosphite ester include those which correspond to the above-listed phosphate esters and acid phosphate esters.

The above-mentioned esters may form amine salts with a mono-, di- or trisubstituted amine represented by the formula (II) below.

10



(II)

15 (where R denotes a C₃-30 alkyl or alkenyl group, a C₆-30 aryl group or aralkyl group, or a C₂-30 hydroxyalkyl group; and n is 1, 2, or 3. Two or more R's may be the same or different. The alkyl or alkenyl group may be straight, branched, or cyclic.)

20 Examples of the monosubstituted amine include butylamine, pentylamine, hexylamine, cyclohexylamine, octylamine, laurylamine, stearylamine, oleylamine, and benzylamine. Examples of the disubstituted amine include dibutylamine, dipentylamine, dihexylamine, dicyclohexylamine, dioctylamine, dilaurylamine, distearylamine, dioleylamine, dibenzylamine, stearyl monoethanolamine, decyl monoethanolamine, hexyl monopropanolamine, benzyl monoethanolamine, phenyl monoethanolamine, and tolyl monoethanolamine. Examples of the trisubstituted amine include tributylamine, triphenylamine, trihexylamine, tricyclohexylamine, trioctylamine, trilaurylamine, tristearylamine, 25 trietylamine, tribenzylamine, dioleyl monoethanolamine, dilauryl monopropanolamine, dioctyl monoethanolamine, dihexyl monopropanolamine, dibutyl monopropanolamine, oleyl diethanolamine, stearyl dipropanolamine, lauryl diethanolamine, octyl dipropanolamine, butyl diethanolamine, benzyl diethanolamine, phenyl diethanolamine, tolyl dipropolamine, xylol diethanolamine, triethanolamine, and tripropanolamine.

25 Of these phosphorus-based extreme pressure additives, tricresyl phosphate and amine salts of alkyl or alkenyl acid phosphate ester are preferable because of their good extreme pressure properties and wear resistance.

30 Some of the above-listed phosphorus-based extreme pressure additives will serve as the components (A) and (B) because they contain both sulfur and phosphorus in the molecule. They include thiophosphate ester, acid thiophosphate ester and amine salts thereof, and sulfurized terpenes.

The above-mentioned phosphorus-based extreme pressure additives may be used alone or in combination with one another. Their amount should be 0.05-5 wt%, preferably 0.1-3 wt%, of the total amount of the lubricant composition. An amount less than 0.05 wt% is not enough for satisfactory extreme pressure properties and wear resistance. An amount exceeding 5 wt% leads to sludge and rust.

35 The lubricant composition of the present invention contains an alkaline earth metal-based detergent as the component (C), which is not specifically restricted so long as it has alkaline earth metal in the molecule and is capable of dissolving or uniformly dispersing in the base oil to exhibit the extreme pressure properties and good wear resistance. It includes, for example, sulfonate, phenate, salicylate, and phosphate of alkaline earth metal. Calcium phenate is desirable because of its ability to improve the coefficient of friction.

40 The alkaline earth metal-based detergent should preferably have a base number in the range of 80-350 mg KOH/g. With a base number lower than specified, it does not produce the desired effect. With a base number higher than specified, it has an adverse effect on wear resistance. A preferred base number ranges from 100 to 280 mg KOH/g.

45 The alkaline earth metal-based detergents may be used alone or in combination with one another. Their amount should be 0.05-8 wt%, preferably 0.1-4 wt%, of the total amount of the lubricant composition. An amount less than 0.05 wt% is not enough for satisfactory effect. An amount exceeding 8 wt% leads to incomplete dissolution in the base oil.

The lubricant composition of the present invention may be incorporated with the following optional additives in an amount not harmful to the object of the present invention. Antioxidant, ashless dispersant, viscosity index improver, pour point depressant, rust preventive, metal deactivator, anti-foaming agent, surface active agent, and coloring agent.

50 The antioxidant fall into three categories as follows.

(1) Hindered phenol.

55 4,4'-bis(2,6-di-t-butylphenol),
4,4-bis(2-methyl-6-t-butylphenol),
4,4'-bis(2-methyl-6-t-butylphenol),
2,2'-methylenebis(4-ethyl-6-t-butylphenol),
2,2'-methylenebis(4-methyl-6-t-butylphenol),
4,4'-butyldienebis(3-methyl-6-t-butylphenol),
4,4'-isopropylidenebis(2,6-di-t-butylphenol),

2,2'-methylenebis(4-methyl-6-nonylphenol),
 2,2'-isobutylidenebis(4,6-diemethylphenol),
 2,2'-methylenebis(4-methyl-6-cyclohexylphenol),
 5 2,6-di-t-butyl-4-methylphenol,
 2,6-di-t-butyl-4-ethylphenol,
 2,4-dimethyl-6-t-butylphenol,
 2,6-di-t-amyl-p-cresol,
 2,6-di-t-butyl-4-(N,N'-dimethylaminophenol),
 10 4,4'-thiobis(2-methyl-6-t-butylphenol),
 4,4'-thiobis(3-methyl-6-t-butylphenol),
 2,2'-thiobis(4-methyl-6-t-butylphenol),
 bis (3-methyl-4-hydroxy-5-t-butylbenzyl)sulfide,
 bis (3,5-di-t-butyl-4-hydroxybenzyl)sulfide,
 n-octadecyl-3-(4-hydroxy-3,5-di-t-butylphenyl)propionate, and
 15 2,2'-thio[diethyl-bis-3-(3,5-di-t-butyl-4-hydroxyphenyl)propionate].

Those of bisphenol type and ester group-containing phenol type are preferable.

20 (2) Amine.
 Monoalkyldiphenylamine such as mono{octyldiphenylamine} and monononyldiphenylamine.
 Dialkyldiphenylamine such as 4,4'-dibutyldiphenylamine, 4,4'-dipentylidiphenylamine, 4,4'-dihexyldiphenylamine, 4,4'-diheptyldiphenylamine, 4,4'-dioctyldiphenylamine, and 4,4'-diononyldiphenylamine. Polyalkyldiphenylamine such as tetrabutyldiphenylamine, tetrahexyldiphenylamine, tetra{octyldiphenylamine}, and tetra{nonyldiphenylamine}. Naphthylamine such as α -naphthylamine, phenyl- α -naphthylamine, butylphenyl- α -naphthylamine, pentylphenyl- α -naphthylamine, hexylphenyl- α -naphthylamine, heptylphenyl- α -naphthylamine, octylphenyl- α -naphthylamine, nonylphenyl- α -naphthylamine, and other alkyl-substituted-phenyl- α -naphthylamines.

Of these examples, dialkyldiphenylamine and naphthylamine are preferable.

30 (3) Zinc dialkyldithiophosphate (ZnDTP).

Zinc diamylthiophosphate, zinc dibutylthiophosphate, and zinc di(2-ethylhexyl)dithiophosphate.
 Examples of the ashless dispersant include succinimide, polybutenyl succinimide, boron-containing succinimide, benzylamine, boron-containing benzylamine, succinate ester, and amide of fatty acid or mono- or dibasic carboxylic acid represented by succinic acid.
 Examples of the viscosity index improver include polymethacrylate, dispersed polymethacrylate, olefin copolymer (such as ethylene-propylene copolymer), dispersed olefin copolymer, and styrene copolymer (such as styrene-diene (hydrogenated) copolymer). Examples of the pour point depressant include polymethacrylate.
 The rust preventive includes, for example, alkenyl succinic acid and partial ester thereof. The metal deactivator includes, for example, benzotriazole, benzimidazole, benzothiazole, and thiaziazole. The anti-foaming agent includes, for example, dimethylpolysiloxane and polyacrylate. The surface active agent includes, for example, polyoxyethylene alkylphenyl ether. These additives are usually incorporated in an amount of 0.01-10 wt% of the total amount of the composition.

The lubricant composition of the present invention is capable of keeping the coefficient of friction higher than 0.10 for a long period of time; therefore, it is capable of torque transmission in large capacities and it is particularly suitable for transmission of metal belt type.

To further illustrate the invention, and not by way of limitation, the following examples are given.

Examples 1 to 3 and Comparative Examples 1 to 4

50 In each example, a lubricant composition was prepared from a paraffin mineral oil (as the base oil) and additives (shown in Table 1) by stirring at 60°C.
 The resulting lubricant composition was measured for the coefficient of friction and the length of the time through which the coefficient of friction was maintained by using a pin-on-disc tester in the following manner. The results are shown in Table 1.
 Conditions for the pin-on-disc tester:

Amount of oil: 600 ml
 Temperature of oil: 130°C

Slip speed: 1200 mm/sec
 Surface pressure: 20 kgf/cm²
 Pin: S45C
 Disc: SCM420
 Duration: 240 minutes

The coefficient of friction was measured after 240 minutes. The length of the time (in minutes) through which the coefficient of friction higher than 0.10 was maintained was measured.

10 Table 1

Lubricant composition (wt%)		Example			Comparative Example			
		1	2	3	1	2	3	4
	Paraffin base oil	92.0	91.5	90.5	92.0	92.0	92.0	92.5
(A)	Sulfurized oil	0.5	—	1.0	1.0	1.0	—	1.0
	Thiotorpene	—	1.0	—	—	—	—	—
(B)	Tricresyl phosphate	0.5	—	—	1.0	—	1.0	—
	Acid phosphate ester amine	—	0.5	1.0	—	—	—	—
(C)	Calcium sulfonate	—	—	1.0	—	—	1.0	—
	Calcium phenate	1.0	1.0	—	—	1.0	—	—
Others	ZnDTP	—	—	0.5	—	—	—	0.5
	Succinimide dispersant	1.0	1.0	1.0	1.0	1.0	1.0	1.0
	Polymethacrylate	5.0	5.0	5.0	5.0	5.0	5.0	5.0
	Coefficient of friction	0.14	0.14	0.12	0.09	0.06	0.09	0.07
	Duration of coefficient of friction (min)	240<	240<	240<	20	15	60	60

40 Note Table 1

Base oil: kinematic viscosity, 4.5 cSt (100°C); %C₄, 0.1; pour point, 17.5°C

Sulfurized oil: sulfurized lard (9 wt% sulfur)

Thiotorpene: reaction product of phosphorus pentasulfide and pinene (10 wt% sulfur)

Acid phosphate ester amine: amine salt of dilauryl acid phosphate

Calcium sulfonate: base number 280 mg KOH/g

Calcium phenate: base number 250 mg KOH/g

ZnDTP: zinc di-C₄₋₆-alkyldithiophosphate

Succinimide dispersant: polybutenylsuccinimide

Polymethacrylate: molecular weight 40,000

55 It is noted from Table 1 that the samples in Comparative Examples decrease in the coefficient of friction (and hence become poor in torque transmission) more rapidly than the samples in Examples.

Comparative Example 5

The pin-on-disc test as mentioned above was conducted on a commercial automatic transmission fluid (ATF) equivalent to Dexiron III®. Seizure took place one minute after the start of the test.

5 The above-mentioned results suggest that the lubricant composition of the present invention maintains the coefficient of friction higher than 0.10 for a long period of time, so that it is capable of transmitting a large capacity of torque when it is applied to an continuous variable transmission. It is suitable for a transmission of metal belt type.

Industrial Applicability

10 The lubricant composition of the present invention is superior in wear resistance and extreme pressure properties and keeps the coefficient of friction high for a long period of time, so that it is capable of transmitting a large capacity of torque when it is applied to an continuous variable transmission. It is particularly suitable for a transmission of metal belt type.

15 Claims

1. A lubricant composition for continuous variable transmissions which comprises a base oil, a sulfur-based extreme pressure additive (A), a phosphorus-based extreme pressure additive (B), and an alkaline earth metal-based detergent (C).

2. A lubricant composition for continuous variable transmissions as defined in Claim 1, wherein the sulfur-based extreme pressure additive is at least one species selected from sulfurized oils and fats, thiocarbamates, and thioterpenes.

25 3. A lubricant composition for continuous variable transmissions as defined in Claim 1, wherein the phosphorus-based extreme pressure additive is at least one species selected from tricresyl phosphate and amine salts of alkyl or alkenyl acid phosphate ester.

30 4. A lubricant composition for continuous variable transmissions as defined in any of Claims 1 to 3, wherein the alkaline earth metal-based detergent is calcium phenate.

5. A lubricant composition for continuous variable transmissions as defined in any of Claims 1 to 4, wherein the amount of components (A), (B), and (C) based on the amount of the lubricant composition is 0.05-5 wt%, 0.05-5 wt%, and 0.05-8 wt%, respectively.

35 6. A lubricant composition for continuous variable transmissions as defined in any of Claims 1 to 5, wherein the continuous variable transmission is of metal belt type.

40 7. A method for lubricating continuous variable transmissions with the lubricant composition defined in any of Claims 1 to 6.

8. A method for lubricating continuous variable transmissions, designed for performing continuous speed change and torque transmission simultaneously, with the lubricant composition defined in any of Claims 1 to 6.

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INTERNATIONAL SEARCH REPORT

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A. CLASSIFICATION OF SUBJECT MATTER
 Int. C16 C10M163/00, C10M135/18, C10M135/20, C10M151/00,
 C10M135/04, C10M137/04, C10M137/08, C10M159/20, C10N40:04
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B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

Int. C16 C10M163/00, C10M135/18, C10M135/20, C10M151/00,
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Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	JP, 7-3283, A (Ethyl Petroleum Additives, Ltd.), January 6, 1995 (06. 01. 95) & US, 5492638, A & EP, 620268, A1	1 - 8
X	JP, 6-336593, A (Tonen Corp.), December 6, 1994 (06. 12. 94) & EP, 707623, A1	1 - 8
X	JP, 6-501516, A (The Lubrizol Corp.), February 17, 1994 (17. 02. 94) & EP, 537338, A1	1 - 8

 Further documents are listed in the continuation of Box C. See patent family annex.

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(54) Lubricating oil composition for power control.

(57) Disclosed is a lubricating oil composition for power control, comprising (A) a base oil having a kinematic viscosity at 100 °C of 1 to 80 cSt, (B) 0.1 to 10% by weight (based on the total weight of the composition, the same shall apply hereinafter) of at least one kind of sulfur-containing compound selected from the group consisting of zinc dithiophosphate, sulfurized oils and fats and sulfurized olefin, (C) 0.1 to 10% by weight of alkaline earth metal based detergent-dispersant, and (D) 0.05 to 5% by weight of partial esters of polyhydric alcohols and/or succinimide.

Said composition is excellent in extreme-pressure property, antiwear property, and metal fatigue life, and has also a good initial frictional characteristics. In addition, the frictional characteristics hardly change with the time, and said composition is excellent in stability against oxidation, and in corrosion resistance. Further, since said lubricating oil compositions include those excellent in heat-resistance, they show an excellent lubricity for a long period when used as gear oil for automobiles, to extend the cycle time for oil replacement.

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LUBRICATING OIL COMPOSITION FOR POWER CONTROL

BACKGROUND OF THE INVENTION

5 1. Field of the Invention

The present invention relates to a lubricating oil composition for power control, and more specifically to a lubricating oil composition which is excellent in frictional characteristics, extreme-pressure properties, antiwear properties and the like, and which is suitable as a lubricating oil for transmission gears, differential gears, hypoid gears of automobiles used at a high temperature for a long period.

15 2. Description of the Related Arts

Generally, as lubricating oils for power control mechanisms, including oils for transmission gears of automobiles, conventionally used ones have mainly been the lubricating oils in which anti-scoring properties to the tooth surfaces of gears are regulated by controlling the amount of sulfur-phosphorus based extreme-pressure agent to be added.

In recent years, however, automobiles have shown a marked trend to be higher-graded or distinctly classified. With such a trend, a smooth and comfortable operation in driving, that is, a favorable operation feeling has been regarded as important. In order to improve the operation feeling in driving, it is desired that the frictional property between the synchronizer ring and the gear cone in the synchromesh mechanism is so favorable (that means, ratio of coefficient of static friction (μ_0) to coefficient of kinematic friction (μ_k), i.e., μ_0/μ_k is small, and said ratio hardly changes with time), that the transmission can be operated smoothly. The use of conventional gear oils containing sulfur-phosphorus based extreme-pressure agent has frequently resulted in poor frictional property, and operation feeling has not been favorable.

In view of these points, various lubricating oil compositions have been proposed for improving the operation feeling (Japanese Patent Application Laid Open No. 192495/1987, No. 61090/1988, and No. 61091/1988). However, they have met problems in corrosion of lubricated portions and in the stability of the whole composition.

On the other hand, in the transmission of large-sized trucks in which oil temperature amounts to 150°C or more, insufficient heat-resistance has caused deterioration, corrosion of metals, formation of sludge and other problems, various troubles such as abrasion or damage of teeth surface of gears, damages or seizure of bearings, and further, slip out of gear in some cases. Phosphoric acid esters and phosphorous acid esters which are included in phosphorus extreme pressure agents are afraid to accelerate said deterioration or corrosion, and accordingly the use of them has an inconvenience that lubricating oil must be replaced in a short period.

40 SUMMARY OF THE INVENTION

An object of the present invention is to provide a lubricating oil composition which is excellent in antiwear properties, and extreme-pressure properties, and in addition, favorable in heat-resistance, particularly a lubricating oil composition of a new type containing no phosphorus extreme-pressure agent.

Another object of the present invention is to provide a lubricating oil composition for power control which is excellent in anti-wear properties, and extreme-pressure properties, and at the same time favorable in the operation feeling mentioned above.

The present invention provides a lubricating oil composition for power control comprising:

- (A) a base oil having a kinematic viscosity at 100°C of 1 to 80 cSt,
- (B) 0.1 to 10% by weight (based on the total weight of the composition; the same shall apply hereinafter) of at least one kind of sulfur-containing compound selected from the group consisting of zinc dithiophosphate, sulfurized oils and fats, and sulfurized olefins,
- (C) 0.1 to 10% by weight of alkaline earth metal based detergent-dispersant, and
- (D) 0.05 to 5% by weight of partial ester of polyhydric alcohol and/or succinimide.

DESCRIPTION OF PREFERRED EMBODIMENTS

- Various oils can be used for the base oil as component (A) of the present invention as long as they have a kinematic viscosity at 100 °C of 1 to 80 cSt, preferably 3 to 50 cSt.
- 5 Thus, mineral oil-based lubricating oil fraction and synthetic lubricating oil are also available for this component. An example of the mineral oil-based lubricating oil fraction is a purified oil obtained by purifying a distillate oil with a conventional method, said distillate oil having been obtained by atmospheric distillation of a paraffin based crude oil, a naphthene based crude oil, or an intermediate based crude oil, or by vacuum distillation of a residual oil resulting from the atmospheric distillation. Examples of the synthetic lubricating oils are poly-alpha-olefins, polybutenes, dibasic acid esters, polyglycols, hindered esters, alkylbenzenes, polyethers and the like.
- 10 Regarding the sulfur-containing compounds as component (B) of the present invention, zinc dithiophosphate, sulfurized oils and fats, and sulfurized olefins are used singly or in combination. Therein, as zinc dithiophosphate (ZnDTP), various ones conventionally used including zinc diisopropyl-dithiophosphate, zinc di-2-ethylhexylidithiophosphate, zinc di-n-butylidithiophosphate, zinc di-n-amylidithiophosphate, zinc di-n-hexylidithiophosphate, zinc di-n-octylidithiophosphate, and zinc dinonylphenylidithiophosphate can be used.
- 15 When alkaline earth metal based detergent-dispersant as component (C) has a base number of 200 mg KOH/g or more, branched alkyl ZnDTP and/or alkylphenyl ZnDTP preferably used. In that case, it is preferred that branched alkyl ZnDTP and/or alkylphenyl ZnDTP may occupy more than half the quantity of whole ZnDTP used there. Herein preferable examples of the branched alkyl ZnDTP or alkylphenyl ZnDTPs are those of which branched alkyl groups or alkylphenyl groups have 3 to 20 carbon atoms, specifically, ZnDTPs of which branched alkyl groups are iso-propyl groups, 2-ethylhexyl groups, or alkylphenyl groups are nonylphenyl groups, as main constituent.
- 20 When alkaline earth metal based detergent-dispersant as component (C) has a base number of less than 200 mg KOH/g, primary ZnDTP shows a superior stability against oxidation, and is favorably used accordingly. In that cases it is preferred that primary ZnDTP may occupy more than half the quantity of whole ZnDTP used there. Herein preferable examples of the primary ZnDTPs are those of which alkyl groups are primary and have 3 to 20 carbon atoms, specifically, ZnDTPs of which alkyl groups are n-butyl groups, n-amyl groups, n-hexyl groups, or n-octyl groups as main constituent.
- 25 The sulfurized oils and fats are obtained by reacting sulfur or sulfur-containing compound with oils and fats (lard, soy bean oil, rice bran oil and the like) or terpene. Sulfur content of said oil is not critical, but preferably 5 to 70% by weight.
- 30 Sulfurized olefin is obtained by reacting olefin having 2 to 15 carbon atoms (propylene, isobutylene, diisobutene and the like) with sulfur or sulfur-containing compounds, or by sulfurizing an olefin polymer (polypropylene, polyisobutylene and the like), and its sulfur content is preferably 20 to 70% by weight.
- 35 In the composition of the present invention, the proportion of the abovementioned component (B) is 0.1 to 10% by weight, preferably 0.5 to 5% by weight, and more preferably 0.5 to 3% by weight based on the total weight of the composition. If the proportion is less than 0.1% by weight, effects of addition is hardly apparent. If it is in excess of 10% by weight, effects corresponding to the added amount are not seen, but on the contrary, stability against oxidation or metal corrosion resistance and other properties might be lowered.
- 40 As component (C) of the present invention, alkaline earth metal based detergent-dispersants having the most suitable base number for each purpose are used. Specified examples are the sulfonates, phenates (phenolates), and salicylates of calcium, barium or magnesium. Of these salts, the most suitable are salts of calcium. Besides them, also phosphonates or naphthenates can be used.
- 45 Alkaline earth metal based detergent-dispersant having a base number of 200 mg KOH/g or more, more specifically 250 to 500 mg KOH/g, has the effect to raise the coefficient of kinematic friction (μ_k) of synchronous μ -V characteristics (frictional characteristics) in the synchromesh mechanism. With the use of alkaline earth metal based detergent-dispersant used here having a base number of less than 200 mg KOH/g, said coefficient of kinematic friction (μ_k) becomes lowered, which may result in increase in μ_0/μ_k to make frictional characteristics unfavorable. The proportion of alkaline earth metal based detergent-dispersant in that case is 0.1 to 5% by weight, preferably 0.5 to 3% by weight based on the total weight of the composition. If it is less than 0.1% by weight, effect of addition is not shown sufficiently, and if it is in excess of 5% by weight, amount of abrasion becomes unfavorably increased.
- 50 With the use of the alkaline earth metal based detergent-dispersant having a base number of less than 200 mg KOH/g, particularly 3 to 180 mg KOH/g, an effect to improve heat resistance appears. Therein, if the base number of alkaline earth metal based detergent-dispersant is 200 mg KOH/g or more, metal

fatigue (service life of bearing is shortened) may occur when used at a high temperature for a long term. In that case, the proportion of alkaline earth metal based detergent-dispersant is 0.1 to 10% by weight, preferably 1 to 8% by weight based on the total weight of the composition. If the proportion is less than 0.1% by weight, the effect of addition is not shown sufficiently, and if it is in excess of 10% by weight, any improvement in effect corresponding to the amount added is not seen, but amount of abrasion is unfavorably increased.

In the present invention, further, a partial ester of a polyhydric alcohol and/or succinimide are/is used as component (D). Therein, partial esters of polyhydric alcohols include various kinds such as monoesters and diesters of dihydric to hexahydric alcohols. Examples of them are partial esters of alcohols including glycol, 10 glycerol, trimethylol propane, pentaerythritol, sorbitol and the like, combined with an organic acid residue having 8 to 30 carbon atoms (lauric acid residue, stearic acid residue, oleic acid residue, behenic acid residue and the like). Preferred examples are specifically sorbitan monolaurate, sorbitan dilaurate, sorbitan monooleate, sorbitan dioleate, sorbitan monostearate, sorbitan distearate, sorbitan monobehenate, sorbitan dibehenate, glycerol monolaurate, glycerol monooleate, and the like.

15 Said partial esters of polyhydric alcohols are effective components in improving frictional characteristics of lubricating oil composition, and shows a remarkable effect particularly when used in combination with said alkaline earth metal based detergent-dispersant as component (C) having a base number of 200 mg KOH/g or more. In order to draw forth said effect fully, the proportion of the abovementioned partial ester should be 0.05 to 3% by weight, preferably 0.1 to 2% by weight based on the total weight of the 20 composition. If the proportion is less than 0.05% by weight, the effect by addition is not developed sufficiently, and if it is in excess of 3% by weight, stability against oxidation and water separation properties will receive a bad influence.

25 As the succinimides, various ones including alkenyl succinimide and alkyl succinimide can be used. Similarly reaction products and derivatives thereof obtained by reacting a boron compound (boric acid, boric acid salt, boric acid ester and the like) with alkenyl succinimide, or alkyl succinimide can be used. Among them, preferred one is alkenyl succinimide containing an alkenyl group having 15 to 500 carbon atoms, for example, polybutenyl succinimide having a molecular weight of 200 to 5000.

30 Said succinimide is an effective component to improve the heat-resistance of lubricating oil composition. It shows a remarkable effect particularly when used in combination with alkaline earth metal based detergent-dispersant having a base number of less than 200 mg KOH/g as component (C). In order to demonstrate said effect fully, the proportion of the succinimide should be 0.1 to 5% by weight; preferably 0.5 to 3% by weight based on the total weight of the composition. If said proportion is less than 0.1% by weight, effect by addition is not shown sufficiently, and if it is in excess of 5% by weight, no improvement in effect can be expected.

35 The lubricating oil composition of the present invention comprises components (A) to (D) mentioned above as inevitable components, and if necessary, each an appropriate amount of various additives such as ashless dispersants, extreme-pressure agents, viscosity index improvers, pour point depressants, defoaming agents and others can be compounded.

40 The lubricating oil composition of the present invention has sufficient extreme-pressure properties, anti-wear properties, metal fatigue life, and also favorable initial frictional characteristics, which means that the ratio of static frictional coefficient/kinematic frictional coefficient is small, and the shock by gear shifting is so moderate as to offer a comfortable operation at driving an automobile. In addition, changes with time of frictional characteristics are small, and said lubricating oil composition is excellent in stability against oxidation and corrosion resistance.

45 Further, since the lubricating oil composition of the present invention includes those having a high heat resistance, it shows an excellent lubricity over a long period when applied as gear oils for automobiles, and accordingly the period of replacing oil can be extended.

50 Consequently, the composition of the present invention is useful as a lubricating oil for power control including gear oil for automobiles, lubricating oil for the parts having a wet clutch or a wet blake, industrial gear oils and the like.

The present invention is described in greater detail with reference to the following examples and comparative examples.

55 Examples 1 to 3 and Comparative Examples 1 to 4

As shown in Table 1, various kinds of additives were compounded in prescribed ratios to paraffin based lubricating base oil (150 neutral), to prepare lubricating oil compositions.

The resulting lubricating oil compositions were subjected to performance tests as follows. The results are given in Table 1.

5 SAE (Society of Automobile Engineers) No. 2 Friction Test

With the use of SAE No. 2 Tester (manufactured by Greening Association Inc., USA), frictional characteristics were evaluated under the following condition for experiment.

10 (Test Condition)

Disk: brass synchromesh ring

Plate: steel gear cones

15 Revolution of motor: 1500 rpm

Piston pressure: 50 kg/cm²

Oil temperature: 60 °C

The coefficient of kinematic friction (μ_{1000}) at 1000 rpm under the above test condition, and the coefficient of static friction (μ_0) at stopping were measured, and μ_0/μ_{1000} was calculated.

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Antiwear properties

In accordance with ASTM D 2714, measurement was done according to the following condition.

25 Block: brass (JIS third class)

Ring: SUJ 2

Oil temperature: 60 °C

Load: 200 pounds

Revolution number: 500 rpm

30 Period: 15 minutes

Examples 4 and 5 and Comparative Examples 5 to 7

35 As shown in Table 2, various kinds of additives were compounded in the prescribed ratio to paraffin-based lubricating base oil (kinematic viscosity at 100 °C: 20 cSt), to prepare lubricating oil compositions.

Lubricating oil compositions thus obtained were subjected to the tests shown below. The results are shown in Table 2.

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Copper Plate Corrosion Test

Test was carried out in accordance with JIS K 2513 on the condition of the temperature of 130 °C and the period of 3 hours.

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Oxidation Stability Test

50 Test was carried out in accordance with JIS K 2514. 3.1, on the condition that the test temperature was 150 °C, and test period was 96 hours. The amount of undissolved portion denotes the amount of undissolved n-pentane determined according to ASTM D 893, method B.

Shell Four-Ball Test

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Test was carried out in accordance with ASTM D 4172. Each of the test samples was an oil degraded in the abovementioned Oxidation Stability Test.

Conditions: 1200 rpm, 60 min., 40 kg/cm²,

oil temperature: 75 °C

Scrolling Four-Ball Test

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This was carried out in accordance with the method described in Japanese Patent Application Laid Open No. 147263/1984.

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Table 1 (Compositions of test samples (wt%) and the results)

Component	Example			Comparative Example		
	1	2	3	1	2	3
Base Oil	90	90	91	91.5	92	91.5
ZnDTP	1.5	2.0	—	1.5	—	1.5
Sulfurized Fats and Oils*1	0.5	—	—	0.5	—	—
Sulfurized Olefin*2	—	—	1.0	—	—	0.5
Ca Sulfonate*3	1.5	—	1.5	—	1.5	1.5
Mg Sulfonate*4	—	1.5	—	—	—	—
Sorbitan Monoleate	0.5	—	—	0.5	0.5	—
Sorbitan Dioleate	—	—	0.5	—	—	—
Glycerol Monooleate	—	0.5	—	—	—	—
Succinimide	1.0	1.0	1.0	1.0	1.0	—
Polymer*5	5.0	5.0	5.0	5.0	5.0	5.0

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Table 1 (continued)

Results	Example				Comparative Example
	1	2	3	4	
SAE No.2 Test	0.074	0.082	0.081	0.074	0.088
μ_0	0.069	0.068	0.074	0.057	0.062
μ_{1000}	1.07	1.21	1.09	1.30	1.44
μ_0/μ_{1000}					1.41
Antiwear Property (mg)	32	30	28	32	150
					32
					35

- *1. Sulfurized lard (Sulfur Content: 10 wt%)
- *2 Reaction product of Isobutylene and Sulfur-Hydrogen Sulfide Mixture
- *3 Base number: 300 mg KOH/g
- *4 Base number: 300 mg KOH/g
- *5 Polymethacrylate (weight average molecular weight: 100000)
- *6 A commercial lubricating oil of GL-4 class containing Sulfur-Phosphorus based Extreme-Pressure Agent

56 50 45 40 35 30 25 20 15 10 5

Table 2

Component	Base Oil	(wt%)	Example			Comparative Example	
			4	5	6	7	
ZnDTP *1	(wt%)	92.5	93.5	97.5	94.0	Commercial Products*5	
Ca Sulfonate *2	(wt%)	1.5	2.0	1.5	—	Commercial Products*5	
Ba Sulfonate *3	(wt%)	5.0	—	—	5.0	Commercial Products*5	
Succinimide *4	(wt%)	—	3.0	—	—	Commercial Products*5	
Results of Tests		1.0	1.5	1.0	1.0	Commercial Products*5	
Oxidation Stability Test	Viscosity Ratio (100°C)	1.02	1.01	1.08	1.53	1.63	
	Increase in Total Acid Value	0.1	0.1	1.5	7.5	6.1	
	Undissolved Portion	0.01	0.01	0.01	0.13	1.43	
Shell Four-Ball Test diameter after test (mm)		0.45	0.47	0.88	0.50	0.69	
Scrolling Four-Ball Test (minutes)		62	72	—	—	39	

*1 Zinc Di-n-octyldithiophosphate

5 *2 Base number: 80 mg KOH/g

10 *3 Base number: 160 mg KOH/g

15 *4 Polybutenyl succinimide having a molecular weight of
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20 *5 A commercial lubricating oil of GL-5 class containing
Sulfur-Phosphorus based Extreme-Pressure Agent

25 **Claims**

1. A lubricating oil composition for power control comprising:

(A) a base oil having a kinematic viscosity at 100 °C of 1 to 80 cSt,

(B) 0.1 to 10% by weight (based on the total weight of the composition) of at least one kind of sulfur-containing compound selected from the group consisting of zinc dithiophosphate, sulfurized oils and fats and sulfurized olefin,

(C) 0.1 to 10% by weight of alkaline earth metal based detergent-dispersant, and

(D) 0.05 to 5% by weight of at least one kind of compound selected from the partial esters of polyhydric alcohols and succinimide.

2. A lubricating oil composition for power control comprising:

(A) a base oil having a kinematic viscosity at 100 °C of 1 to 80 cSt,

(B) 0.1 to 5% by weight of at least one kind of sulfur-containing compound selected from the group consisting of zinc dithiophosphate, sulfurized oils and fats and sulfurized olefin,

(C) 0.1 to 5% by weight of alkaline earth metal based detergent-dispersant having a base number of not less than 200 mg KOH/g, and

(D) 0.05 to 3% by weight of a partial ester of polyhydric alcohol.

3. A composition defined in Claim 1 or 2, wherein (A) the base oil is a mineral oil-based lubricating oil fraction or synthetic lubricating oil having a kinematic viscosity at 100 °C of 3 to 50 cSt.

4. A composition defined in Claim 2, wherein the alkaline earth metal based detergent-dispersant has a base number of 250 to 500 mg KOH/g.

5. A composition defined in Claim 1 or 2, wherein the partial ester of polyhydric alcohol is at least one kind of compound selected from the group consisting of sorbitan monolaurate, sorbitan dilaurate, sorbitan monooleate, sorbitan dioleate, sorbitan monostearate, sorbitan distearate, sorbitan monobehenate, sorbitan dibehenate, glycerol monolaurate and glycerol monooleate.

6. A gear oil composition comprising:

(A) a base oil having a kinematic viscosity at 100 °C of 1 to 80 cSt,

(B) 0.1 to 10% by weight of zinc dithiophosphate,

(C) 0.1 to 10% by weight of alkaline earth metal based detergent-dispersant having a base number of less than 200 mg KOH/g, and

(D) 0.1 to 5% by weight of succinimide.

7. A composition defined in Claim 6, wherein (A) the base oil is a mineral oil-based lubricating oil fraction or synthetic lubricating oil having a kinematic viscosity at 100 °C of 3 to 50 cSt.

8. A composition defined in Claim 6, wherein more than half the amount of (B) zinc dithiophosphate is occupied by zinc di-n-alkyl dithiophosphate.

9. A composition defined in Claim 6, wherein (C) alkaline earth metal based detergent-dispersant has a base number of 3 to 180 mg KOH/g.

10. A composition defined in Claim 6, wherein (D) succinimide is an alkenyl succinimide containing an alkenyl group having 15 to 500 carbon atoms.

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DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
X	FR-A-1 485 335 (THE LUBRIZOL CORP.) * Page 1, column 1, line 39 - column 2, line 38; page 5, column 2, lines 12-27; page 6, column 1, lines 17-23; page 7, column 1, lines 13-30; page 9, column 2, lines 1-15; page 14, column 1, lines 11-46; page 16, example 13; page 16, column 2, lines 29-38; claims 1,2f,2g,2k * ---	1,3,6,7 ,10	C 10 M 163/00 C 10 M 169/04 // (C 10 M 163/00 C 10 M 129:76 C 10 M 133:56 C 10 M 135:04 C 10 M 135:06 C 10 M 137:10 C 10 M 159:20)
X	FR-A-2 009 296 (MOBIL OIL CORP.) * Page 4, lines 1-23; page 5, lines 12-38; page 6, lines 24-38; page 8, lines 11-19; page 10, lines 9-19; claims 1,3-6 *	1,3	(C 10 M 169/04 C 10 M 129:76 C 10 M 133:56 C 10 M 135:04 C 10 M 135:06 C 10 M 137:10 C 10 M 159:20)
A	---	2,4,6,7 ,10	C 10 M 129:76 C 10 M 133:56 C 10 M 135:04 C 10 M 135:06 C 10 M 137:10 C 10 M 159:20) C 10 N 10:04 -/-
P,X	EP-A-0 305 538 (IDEMITSU KOSAN CO.) * Page 4, lines 5-24; page 14, lines 1-18; page 16, lines 1-24 *	1,3,5	
A	---	2	TECHNICAL FIELDS SEARCHED (Int. Cl.5)
X	FR-A-2 277 882 (CHEVRON RESEARCH CO.) * Page 4, line 32 - page 5, line 12; page 5, line 26 - page 6, line 8; page 8, lines 23-37; page 10, lines 2-6; page 12, lines 14-17; page 15, lines 9-14; page 17, lines 22-37; page 18, lines 1-28 *	1,3	C 10 M
Y	---	6-10 2,5	
A	---	-/-	
The present search report has been drawn up for all claims			
Place of search	Date of completion of the search		Examiner
THE HAGUE	12-03-1990		HILGENGA K.J.
CATEGORY OF CITED DOCUMENTS		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document	
X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document			



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EUROPEAN SEARCH REPORT

Page 2

Application Number

EP 89 12 2243

DOCUMENTS CONSIDERED TO BE RELEVANT			CLASSIFICATION OF THE APPLICATION (Int. Cl.5)						
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim							
Y	GB-A-1 440 261 (EXXON RESEARCH AND ENG. CO.) * page 2, lines 26-36 * ---	6-10	C 10 N 20:02 C 10 N 40:04						
A	EP-A-0 277 729 (AMOCO CORP.) * Claims 1,8,9; page 9, table I *	1,6,8-10							
A	GB-A-1 152 889 (ESSO RES. ENG. CO.) * Page 1, lines 10-45; page 2, lines 11-20; page 3, example 1 *	1,2,4							
TECHNICAL FIELDS SEARCHED (Int. Cl.5)									
<p>The present search report has been drawn up for all claims</p> <table border="1"> <tr> <td>Place of search</td> <td>Date of completion of the search</td> <td>Examiner</td> </tr> <tr> <td>THE HAGUE</td> <td>12-03-1990</td> <td>HILGENGA K.J.</td> </tr> </table> <p>CATEGORY OF CITED DOCUMENTS</p> <p>X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document</p> <p>T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document</p>				Place of search	Date of completion of the search	Examiner	THE HAGUE	12-03-1990	HILGENGA K.J.
Place of search	Date of completion of the search	Examiner							
THE HAGUE	12-03-1990	HILGENGA K.J.							